Perspectives in Science (2016) 8, 22—24

ScienceDirect

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Introduction to porous spinel for refractory (high temp) material☆

Kumar Saurav*, **Manas Ranjan Majhi**, **Vinay Kumar Singh**

Department of Ceramic Engineering, Indian Institute of Technology (BHU), India

Received 24 December 2015; accepted 26 January 2016
Available online 5 February 2016

KEYWORDS
Ceramics;
Mechanical properties;
Microstructure

Summary The paper examines thermal properties of materials. The transient pulse method was used for specific heat, thermal diffusivity and thermal conductivity determination. Porous MgO was synthesis by heating pellets at 1100 °C for 1 h. The resultant porous MgO was then immersed in 10 mol/L aluminum nitrate solution, dried, and reheated at 1300 °C for 2 h to convert it to spinel. The evaluation was performed with the help of mathematical apparatus used for study of fractal structures properties. The method results from generalized relations that were designed for study of physical properties of fractal structures. As it is shown these relations are in a good agreement with the equations used for the description of time responses of temperature for the pulse input of supplied heat.

Introduction

Among the suitable materials for energy saving purpose, porous ceramics are outstanding because they combine intrinsic ceramic properties, such as chemical inertness and refractoriness, with low thermal conductivity. Besides saving energy, the diminished heat loss to the environment also improves working conditions minimizing the employees' stress as a result of their exposure to high temperatures.

Porous ceramic products cover wide range of advanced ceramic materials, which can be oxides ceramics: alumina, zirconia, or non-oxides ceramics: carbides, borides, nitrides, silicides. The properties of porous ceramic product depend on three main factors: the properties of the ceramic material of which the product is made; the topology (connectivity) and shape of the pores; and the relative density of the product (Salvini et al., 2000; Colombo, 2006; Lyckfeldt and Ferreira, 1998; Park et al., 1997; Sarkar et al., 1999; Studart et al., 2006). Meanwhile, pore formers and other starting materials for porous ceramics used in the conventional methods were first prepared separately and then mixed. Among the techniques used to produce these materials (such as the addition of foaming agents and organic compounds), the pore generation via phase transformation presents key aspects, such as easy processing and the absence of toxic volatiles.

☆ This article belongs to the special issue on Engineering and Material Sciences.

* Corresponding author. Tel.: +91 9621199305; fax: +91 9621199305.
E-mail address: ksaurav.cool@gmail.com (K. Saurav).

http://dx.doi.org/10.1016/j.pisc.2016.01.007

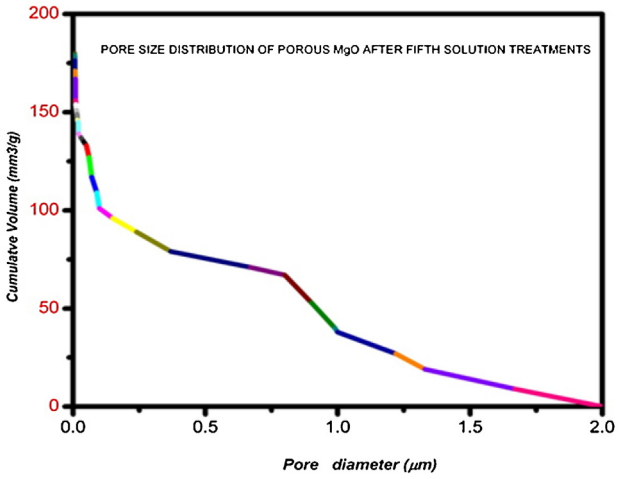

Figure 1 Pore size distribution of porous MgO with fifth solution treatments sample contains (48%) porosity.

Experimental

Fine magnesium oxide (Thomas baker, India, 0.10 mm in diameter and 98% pure) was used as a sintering additive. Ex potato starch (.50 mm, Loba Chemie) and PVA were also added to the starting materials as a pore former. All powders used as pore-formers were previously dried at 100 °C for 24 h. After this step, their density and surface area were measured in Accu Pyc 1330 helium pycnometer and BET equipment (Micrometrics, USA), respectively. Lightly calcined (fine powder) magnesia and Ex potato starch — $C_6H_{10}O_{5n}$ (Loba Chemie, Mumbai) was mixed and then poly vinyl alcohol (fine powder) are mixed. The batches were comprised of porous MgO (60:30:10 — MgO:Starch:PVA) were attrition milled for 3 h and then dried at 100 °C for 24 h. The powders were shaped in pellet (piece of small shot) form using hydraulic pressing machine (uniform pressing) with applied load of 15 ton. The samples were fired at temperatures 1100 °C. The heating rate was maintained at 3 °C/min and soaking period was 2 h. The sintered sample was immersed into the aluminum nitrate solution for 1 h under vacuum, removed, and then dried at room temperature for 24 h. The resultant sample was reheated at 1300 °C for 2 h in air. This solution immersion and reheating treatment was performed up to five times. After each solution treatments are characterized by XRD, SEM. The micrograph in Fig. 1 reveals a homogeneous pore size of less than 2 μm and variation in pore shape and size. At least two different characteristic pore sizes can be identified. The mercury porosimetry measurements confirm that the samples exhibit bimodal pore size distributions, constituted by mesopores with diameter less than 1 μm and by macropores with diameter above 1 μm.

Results and discussion

Spinel formation

The formation of the porous spinel from porous MgO and aluminum nitrate solution after fifth solution treatments was

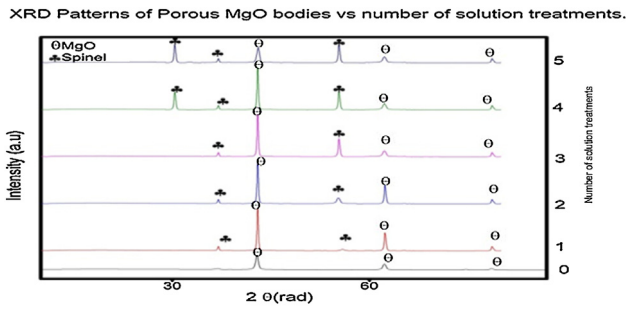

Figure 2 XRD of porous MgO with solution treatments.

studied. Fig. 2 shows diffraction pattern of porous MgO with number of aluminum nitrate solution treatments. Initially after first solution treatment a small peaks of spinel are detected, which indicated that the MgO reacts with alumina and forms spinel. As can be seen, no alumina was detected in any sample, which indicates that the initially precipitated alumina completely reacted with MgO to form spinel during processing. After five solution treatments huge amount of spinel are formed. The ratio of spinel conversion is calculated by weight gain formula according to the chemical reaction. Actually more than 35% spinel are formed after the fifth solution treatments.

Fig. 3 shows an SEM micrograph of a porous MgO sample with fifth solution treatments, a large amount of white ball are developed which indicate huge amount of spinel are formed. This suggests that the alumina platelets were fully covered with the precipitated spinel.

Thermal behavior

The transient pulse method was used for specific heat, thermal diffusivity and thermal conductivity determination. The evaluation was performed with the help of mathematical apparatus used for study of fractal structures properties. Incorporation of porosity into a monolithic material decreases the effective thermal conductivity. Fig. 4 represents the typical time responses of temperature for the step wise of input power. The coefficient f_a (fractal dimension D respectively) of the fractal heat source for every point of the experimental dependence (measured temperature depended on time) was calculated. The fractal heat source characterizes the distribution of the temperature in the specimen in specific time. From Fig. 4 it is evident that for very short time there is the value of the fractal dimension $D \approx 2$ and therefore, the plane heat source is formed.

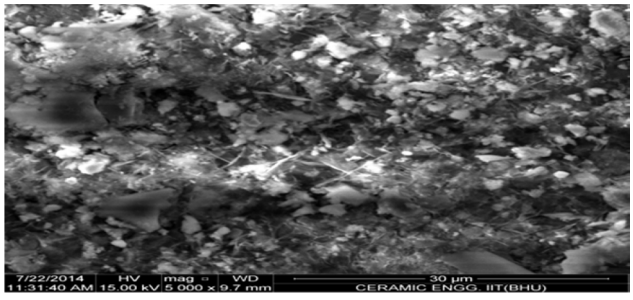

Figure 3 SEM of porous MgO with fifth solution treatments.

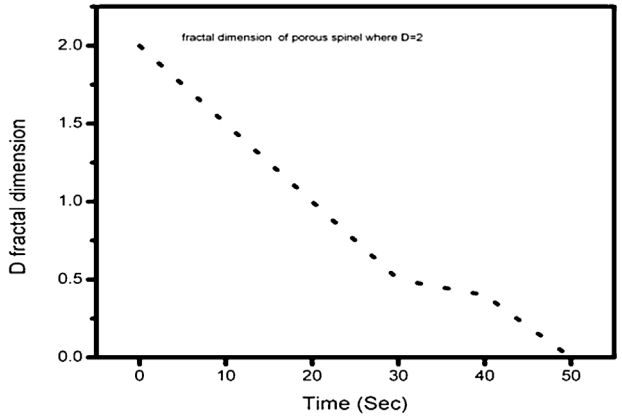

Figure 4 Typical time responses of temperature for the step wise of input power when $D = 2$.

Conclusion

Porous ceramics for applications such as to reduce energy costs, high-temperature insulation can be produced by the decomposition of EX potato known as starch soluble $(C_6H_{10}O_5)n$. In this article, the results of thermal responses to the pulse of supplied heat evaluations are discussed. To interpret the outcomes, the simplified heat conductivity model is used. The coefficient f_a (fractal dimension D respectively) of the fractal heat source for every point of the experimental dependence (measured temperature depended on time) was calculated using the graph.

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